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Gas Chromatographic Analyses of Argo Merchant Oil and Sediment Hydrocarbons at the Wreck Site

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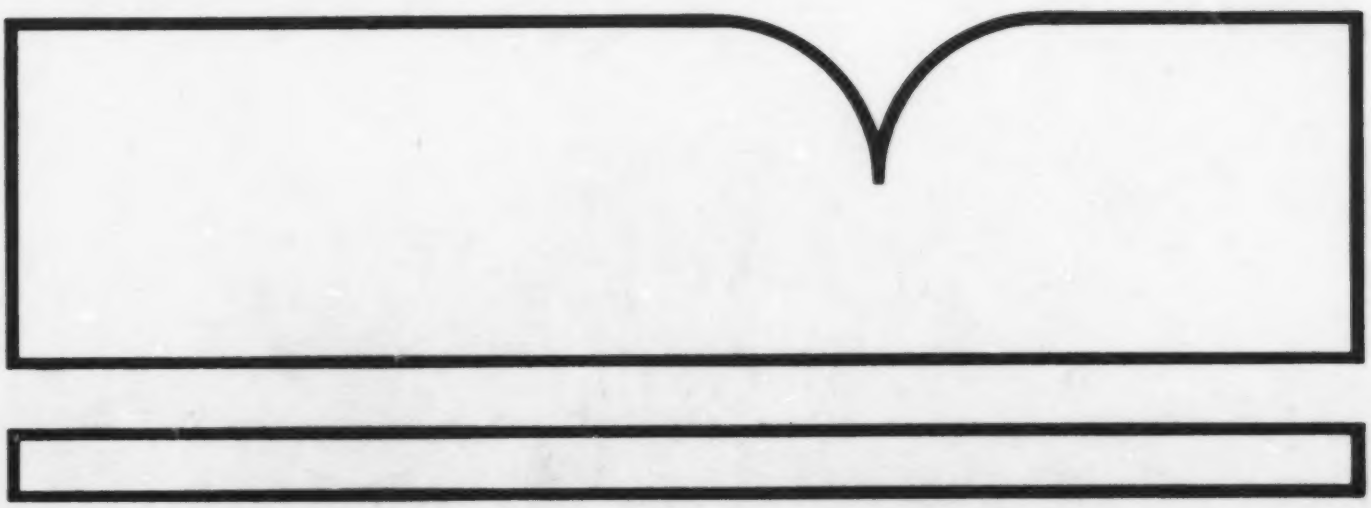

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16. ABSTRACT

Hydrocarbon concentrations were determined in surface sediments in the vicinity of the *Argo Merchant* wreck site. Of the 4000 km² area surveyed, contaminated sediments were found in a 10–15 km² section around the wreck site in February 1977. The contamination was in the form of small tar particles (or oil droplets) inhomogeneously mixed into the sandy sediments by the turbulent shoal tidal currents. Although there was no clear trend of hydrocarbon concentrations as a function of depth, contamination extended at least to 8–13 cm in depth. Stations which were contaminated in February were reoccupied on a cruise in July 1977. Only one station showed any trace of hydrocarbon contamination and this level was significantly lower than found at this site in February.

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labelled food suggests efficient food chain transfer of this organic component.

The situation in *Artemia* is less clear because appreciable radioarsenic taken up from water can be detected with the lipids ($\simeq 23\%$) and as arsenite ($\simeq 56\%$). It may be suggested that an organic component is synthesized, not by this filter feeder itself, but by intestinal microflora. The general conclusion that organic arsenic is synthesized by primary producers and passed along the food chain supports an earlier suggestion by Lunde (1972) that this form of arsenic found in marine animals might be derived from food.

There is at present no information on the exact chemical form of arsenic in lipid extracts; consequently, we have so far refrained from using the term 'lipid arsenic' or 'arsenolipids' since the conversion of arsenate by phytoplankton to a chloroform-extractable derivative could conceivably represent the production of a non-lipid, hydrophobic metabolite. However, it is tempting to speculate that the chemical similarity between arsenic and phosphorus enables arsenate to follow the same metabolic pathways as phosphate in the production of phospholipids. If this is the case then one would expect that polar arseno-analogues of phospholipid precursors and breakdown products are present in biological samples. Accordingly, radioarsenic in water/methanol extracts may represent not only arsenate but also cationic arseno-analogues of substances such as phosphorylethanolamine and phosphorylcholine. This hypothesis would explain why different workers (Penrose, 1975; Lunde, 1977) have reported organic arsenic in both lipid and non-lipid fractions.

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Gas Chromatographic Analyses of *Argo Merchant* Oil and Sediment Hydrocarbons at the Wreck Site

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Hydrocarbon concentrations were determined in surface sediments in the vicinity of the *Argo Merchant* wreck site. Of the 4000 km² area surveyed, contaminated sediments were found in a 10–15 km² section around the wreck site in February 1977. The contamination was in the form of small tar particles (or oil droplets) inhomogeneously mixed into the sandy sediments by the turbulent shoal tidal currents. Although there was no clear trend of hydrocarbon concentrations as a function of depth, contamination extended at least to 8–13 cm in depth. Stations which were contaminated in February were reoccupied on a cruise in July 1977. Only one station showed any trace of hydrocarbon contamination and this level was significantly lower than found at this site in February.

On 15 December 1976, the tanker *Argo Merchant* carrying 28 000 metric tons of No. 6 fuel oil ran aground on Fishing Rip of Nantucket Shoals and soon began to leak her cargo into the surrounding waters. Later, she broke into three parts and the bow section floated off the original wreck site eventually sinking 2.8 km SE of the stern and midsection. In January and early February, 1977, the University of Rhode Island conducted two cruises to the area affected by the surface slick (Nantucket Shoals and Little Georges Bank) to determine if and where the oil had contaminated the benthic environment. Sediments were sampled randomly within a 4000 km² area and screened onboard ship for hydrocarbon content by U.S. Coast Guard chemists using UV

fluorescence spectroscopy. The results of these analyses indicated that sediments were contaminated with *Argo Merchant* oil only around the wreck site (Grose & Mattson, 1977). On the basis of these findings, a detailed sediment survey of the wreck site area was conducted on R/V *Endeavor* cruise 005, 22–27 February 1977. Several of these stations were reoccupied in 22–24 July 1977, 5 months later on F/V *Sideshow* cruise 001.

Fishing Rip Shoal is composed of sandy sediments (fine to coarse sand; Pratt, 1978) with sand ridges perpendicular to the main axis of the shoal. The bottom of the channel to the east of this shoal is gravel bottom containing shell fragments and biological debris. The rotary tidal currents of the area often exceed 3 knots (1.5 m s^{-1}) resulting in erosion and transportation of the sand, and the shallow depth of the water (< 20 m) allows wind mixing to the bottom. These factors produce a very physically active benthic environment. A similar shoal in Vineyard Sound, Massachusetts (Middle Ground) has been shown to be eroded by 2 m every half tidal cycle, and redeposited on the other half cycle (Smith, 1969). The high degree of turbulence associated with Fishing Rip Shoal plays a major role in the findings of this chemical study. Additional studies of the benthic environment of this area after the *Argo Merchant* spill were conducted simultaneously with this investigation. Pratt (1978) examined the sandy sediments for interaction of the oil with the sand and the infaunal species of Fishing Rip Shoal. Brown (1978) examined the macro benthos of the channel to the east of the shoal for histopathological changes. In addition, oil droplet distributions in the water column above the contaminated sediments were also determined (Cornillon, 1978).

The objectives of the present study were as follows: (1) determination of the areal extent of surface sediment contamination by the *Argo Merchant* oil; (2) measurement of the depth of sediment contamination; and (3) evaluation of physical, chemical and/or biological weathering of the oil as a function of time.

Experimental Procedures

Sediment sampling

Sediment samples were collected from the Nantucket Shoals–*Argo Merchant* wreck site area aboard R/V *Endeavor* (EN-005, 22–27 February 1977) and aboard F/V *Sideshow* (SS-001, 22–24 July 1977), Fig. 1. Surface sediment samples (0–15 cm) were collected with a Smith-McIntyre grab sampler. Successive subsamples were taken from various depths and after a small portion (5 ml) of each was removed for on-board screening by Coast Guard personnel, the remainder of each subsample was frozen until later analysis. Sediment cores (13–14 cm each) were obtained on EN-005 with a box corer. Subsamples of each core were taken with plastic core liners and frozen immediately after collection.

Cargo and slick sampling

One cargo sample and two surface slick samples were collected by Dr. Jerome Milgram of Massachusetts Institute of Technology who graciously provided subsamples of these for analysis and information on the collection and storage procedures (Milgram, personal communication, 1977). The *Argo Merchant* cargo oil sample was collected on 19 December 1976. The cargo sample, from the full port tank No. 4, was taken by putting a glass jar into the tank, withdrawing the jar and closing it. The first slick sample (Milgram No. 1) was collected on 19 December 1976, by lowering a bucket from the side of the USCGC *Vigilant*. The sample came from a distance of 2–4 km from the ship and Milgram estimates that it had been on the water for about 1 h. The second sample (Milgram No. 2) was collected on 25 December 1976, at 40°54'N, 68°38.1'W (60 km from the wreck) from a large pancake, also by lowering a bucket from the side of the USCGC *Vigilant*. It had probably been on the water for about 4–5 days. These samples were stored at 0–20°C in glass jars until analysed.

Sediment analysis

Selected sediment samples from EN-005 were chosen for analysis on the basis of two criteria: (1) evidence of measurable or trace quantities of petroleum based on the Coast Guard screening results and/or (2) presence of minute tar particles which appeared on the top of the formalin surface of the biology subsample upon agitation of the sediment. Selection of SS-001 samples were based on (1) indications of contamination found on previous cruises and (2) stations adjacent to contaminated stations of earlier cruises. Occasionally minute tar particles (1–2 mm) were removed from the February samples with tweezers prior to analysis, and the tar particles and sediment were analysed separately.

The sediments were analysed for total hydrocarbons in the range of n-C_{14} to n-C_{34} by a technique described previously (Hoffman & Quinn, 1978). Briefly, 200 g

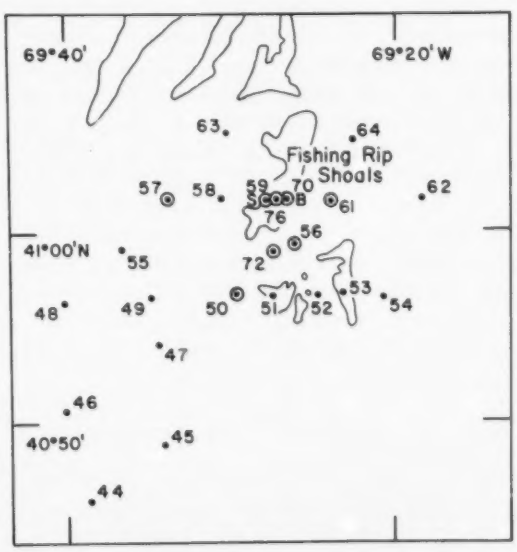

Fig. 1 Station locations of EN-005 (22–27 February 1977), and SS-001 (22–24 July 1977). The "S" (Station 59) is the location of the *Argo Merchant* stern and mid section wrecks and the "B" (Station 70) is the location of the *Argo Merchant* bow section. The circled dots are the stations from which the sediments analysed in this study were collected. The other stations were analysed by the U.S. Coast Guard.

of sediment were extracted by refluxing with a methanolic KOH-toluene mixture. The hydrocarbons were isolated by passing the non-saponifiable fraction through an alumina–silica gel–copper column and analysed by gas–liquid chromatography (GLC). Both packed stainless steel columns (2 m, 2.2 mm i.d., 10% SP-1000) and glass capillary columns (15 m, 0.25 mm i.d., OV-101) were used in this study. The chromatograms were quantified by comparing the areas of the chromatograms with the areas of n-C_{20} and/or n-C_{22} internal standards. The detection limit, after procedural blank correction, was approximately 0.1 μg HC g^{-1} dry weight sediment.

Cargo, slick, and tar particle analysis

After weighing, these samples were dissolved in a mixture of 95% petroleum ether–5% toluene and charged to the Cu-silica gel-alumina column described previously (Hoffman & Quinn, 1978). The hydrocarbon elutant was evaporated to dryness, dissolved in methylene chloride, and analysed by GLC.

Results and Discussion

Areal extent of sediment contamination

The locations where sediment was collected on EN-005 are shown in Fig. 1. Sediments from all of these stations were prescreened by Coast Guard personnel. This study presents detailed GLC analysis only on those samples found to have at least some trace of oil by the Coast Guard technique or those samples from which small tar particles floated out of the corresponding biological samples. A total of 45 subsamples from six EN-005 stations and 15 subsamples from six SS-001 stations were analysed. These later stations are also shown in Fig. 1.

The present data (Table 1) indicate that in February 1977, the sediments were significantly contaminated at three stations (56, 59, 70 – significantly defined as greater than 20 μg HC g^{-1} dry weight, a value ranging from 40 to >200 times higher than the background hydrocarbon concentrations of < 0.5 μg g^{-1}). Two of these stations were located at the wreck site locations: Station 59 at the original wreck site (now the location of the mid and stern sections); and Station 70, the location of the bow section. Station 56 is located 3.2 km SE of the bow section in the channel adjacent to Fishing Rip Shoal. The sediment at 59 and 70 was coarse sand while the sediment at 56 was gravel mixed with crushed shells and mud. Trace levels of hydrocarbon (0.7 to 1.1 μg g^{-1}) were found at Stations 50 and 61 (Station 50 was 4.8 km SW of the werck; Station 61 was 3.2 km E of the wreck). Both of these stations were located in the channel to the S and E of the wreck and the presence of traces of anthropogenic hydrocarbons at these locations could either be from sources other than the *Argo Merchant*, or from the *Argo Merchant* itself. Since the Coast Guard found no evidence of *Argo Merchant* oil at any of the other stations, our findings would indicate that significant sediment contamination in February extended at most 3–4 km from the wreck site in a SE direction. Traces of contamination from an unknown source could be found 3–5 km from the bow in the E and S directions. At most, this would indicate that in February, a 10–15 km^2 area of the sediments was contaminated. It is likely that only a small percentage of this area was significantly contaminated, these areas being limited primarily to the immediate vicinity of the wreck site, especially around the bow section.

TABLE 1
Hydrocarbon concentration in sediments as a function of time.

Station*	Location	μg HC/g dry wt range in February 1977	μg HC/g dry wt range in July 1977
70	Bow section	1.9–122	<0.2–0.6
59	Stern section	<0.1–327	<0.1–0.2
56	3 km SE of wreck	<0.3–21.5	–
61	3 km E of wreck	0.7–1.1	0.4
50	5 km SW of wreck	<0.1–0.8	0.1
72	3 km S of wreck	–	0.2
76	Between bow and stern	–	<0.2–0.4

*Sediment depth = 0–14 cm.

Depth of contamination

An attempt was made to determine the depth of sediment contamination by taking successive subsamples out of Smith-McIntyre grab samples and by examination of box cores. Neither sampling device collected sand any deeper than 13–20 cm, and in the case of gravel bottom stations, a maximum of only 3–4 cm of sediment was collected. No clear trend as a function of depth was noted in the EN-005 sediment samples. An example of this is given in Table 2 which gives the hydrocarbon concentrations as a function of depth at one of the contaminated stations. Samples 59(BC2) and 59(G4) are fairly constant in hydrocarbon concentration as a function of depth, but in the case of 59(BC1), there is an increase with depth in the sediment, and at 59(G1) and 59(G3) there is a decrease with depth.

The probable reasons for this variation are (1) patchiness due to inhomogeneous mixing of minute tar particles into the sand coupled with (2) the highly active

TABLE 2
Hydrocarbon concentration as a function of sediment depth at the stern wreck site in February 1977.

Station	Depth (cm)	μg HC/g dry wt sediment
59(G1)*	0–1	75.9
	1–3	0.5
	3–5	48.6
59(G3)	0–1	327
	1–3	<0.1
	3–5	<0.1
59(G4)	0–1	0.3
	1–3	0.1
	3–5	0.1
59(BC1)	0–3	5.1
	3–8	1.3
	8–13	24.6
59(BC2)	0–4	0.3
	4–9	0.8
	9–14	0.4
Range	0–1	0.3–327
	1–3	<0.1–0.5
	3–5	<0.1–48.6
	8–14	0.4–24.6

*Station designation includes station number (replicate grab or box core sample).

physical nature of the shoals. In this present study, it can only be concluded that contamination of some of the sediment extends at least to 8–13 cm in depth. Clearly, the possibility exists that the contamination could be significantly deeper than this.

Nature of sediment contamination

It was found that at least some of the hydrocarbons in the sediments were associated with small tar particles 1–2 mm in size. In order to find out the approximate percentage of the total hydrocarbons these tar particles represented, particles were physically isolated with spatula and tweezers from four of the samples. The sediments (without their tar particles) and the tar particles were analysed separately. The analytical results indicated that from 80% to greater than 99% of the hydrocarbons in the sediment were associated with these tar particles. Pratt (1978) states that these tar particles were only loosely associated with the sand, and that upon agitation of the sediment, these tar particles could be dislodged completely and float out of the sediment. Indeed, oil droplets were found in the water column over the contaminated sediments at both wreck sites (Cornillon, 1978). Presumably, any oil droplets presented which resulted from the surface slick had been dissipated by the time these water samples were collected. Therefore, the best explanation for the presence of these droplets in the water is the dislodgement of the oil droplets from the sediments by the turbulence on the shoals.

It is also clear that the number and size of the tar particles in any one sediment sample is going to greatly affect the results. In order to minimize this problem, large sediment weights (usually 100–200 g) were used for each analysis in this study. In spite of this precaution, replicate analyses from the same subsample indicate variabilities of a factor of three. Since the uncertainty for the analysis procedure is no greater than 10%, the sediment samples were inhomogeneous even when taken from the same storage container. The patchiness due to inhomogeneous mixing of the tar particles into the sand as a result of the physical activity of the area led to even greater variations from grab sample to grab sample even within 50 m of each other. At Station 70, for example, the upper sediments (0–3 cm depth) in replicate grab and core samples contained 12.8, 10.2, 118, 1.9 and 2.7 $\mu g\ g^{-1}$, a variation over two orders of magnitude. Thus the observed patchiness exists both on micro as well as a macro scale.

Comparison of slick, sediment and tar particle samples with the Argo Merchant *cargo oil*

The chromatographically resolved portion of the hydrocarbon components of the tar particles, sediments and slick samples were compared with the resolved components of the *Argo Merchant* cargo oil, by a simple technique of comparing areas of each resolved component. It should be pointed out that *Argo Merchant* carried two cargos, both No. 6 fuel oils, but only one of the cargos was sampled at the scene. It was this cargo sample that was used for matching purposes in this study. Although these oils were reported to be nearly identical (Grose & Mattson, 1977), some of the following matching experiments could have been affected by differences in the resolved component pattern between the two cargos.

The area of each peak (or resolved component) was expressed in area percent as in the equation:

$$A\%_i = \frac{A_i}{\Sigma A_i} \times 100\%$$

where $A\%_i$ is the area % of the individual resolved component, and A_i is the area in arbitrary units of each individual peak. The individual area percents of each peak of the standard (in this case, the *Argo Merchant* cargo oil sample collected by J. Milgram) are then plotted versus the corresponding area percents (using peaks having the same retention times) of the sample. All the reported peaks were used in the case of packed column chromatographs; however, only the major peaks (with area percentages greater than 1%) were included when glass capillary GLC was used. An example of the result is given in Fig. 2. The area percents of each resolved peak in the chromatogram (Part A of Fig. 2) were plotted versus the corresponding peaks in the cargo sample to yield the resulting graph given in Part B, Fig. 2. Generally, 30–40 peaks were used in each regression analysis. Visually the chromatograms looked similar and linear least squares regression analysis of the individual area percents confirms a strong correlation ($r = 0.90$). A summary of the matching results is given in Table 3. There is a good relationship between *Argo Merchant* oil *vs. Argo Merchant* oil even when analysed on two separate days. There is also a good relationship between the *Argo Merchant* cargo and the Milgram slick samples. Even the 5-day old slick sample showed little weathering, and still had the lighter components of the cutter stock in the slick. (Milgram determined that approximately 20% of the *Argo Merchant* cargo was a light oil added to the No. 6 oil; Grose & Mattson, 1977.) Undoubtedly the cold atmospheric temperature impeded the evaporative losses, and may have reduced the solubility of these components as well. Three out of the

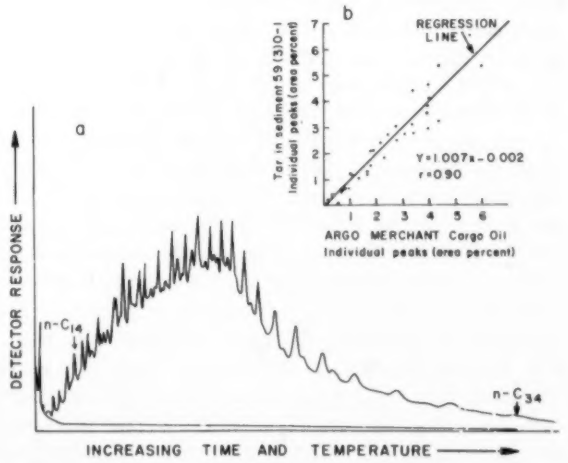

Fig. 2 Part A – Chromatogram (packed column) of tar particles separated from the sediment collected at Station 59, Grab 3, 0–1 cm depth, in February 1977. Part B – Graph of the areas of each resolved hydrocarbon component of this tar particle sample versus the area of the corresponding peaks in the *Argo Merchant* cargo.

TABLE 3
Comparison of environmental samples with *Argo Merchant* cargo oil.

Sample	Correlation coefficient range
Argo Merchant cargo	0.97–0.99
Slick samples (December)	0.91–0.96
Sediment at wreck site, tar particles left in (February)	0.86–0.98
Tar particle samples (February)	0.44–0.90
Sediment at wreck site, tar particles removed (February)	0.74
Sediment station 56 (3 km SE of wreck, February)	0.21
Sediment at wreck site (July)	0.18

four tar particle samples agreed well with the cargo ($r = 0.79–0.90$). The fourth sample ($r = 0.44$) showed signs of weathering or was from a different source. The match with the *Argo Merchant* cargo was good when the sediments with their tar particles were examined; but the relationship was not as good when the sediment alone, without the tar particles, was matched with the cargo. The samples collected further away from the wreck (Station 56) did not match well (the resolved components were highly weathered), and the sediment sample with traces of oil collected in July, 7 months after the spill, did not resemble the cargo oil at all. Clearly more information concerning the source of the hydrocarbons found in these environmental samples could be learned if specific persistent compounds unique to the cargo could be found and studied in the samples.

Variation with time.

On 22–24 July 1977, 5 months after the R/V *Endeavor* cruise 005 in February, and 7 months after the initial spill, further sediment samples from the wreck site areas were collected aboard F/V *Sideshow*. Samples were collected for chemical analysis and biological archiving at all the previously contaminated stations found on EN-005, with the exception of Station 56 (where the July samples contained only cobbles and shell fragments). The hydrocarbon analytical results are given in Table 1. The samples all contained 0.6 *u*g HC g^{-1} or less. Only at one station (Station 70) were traces of anthropogenic hydrocarbons found. As mentioned earlier, the hydrocarbons in this sample did not resemble at all the cargo of the *Argo Merchant*. It more closely resembled patterns reported by Butler *et al.* (1973) and Wade *et al.* (1976) of pelagic tar of the North Atlantic Ocean. It also resembled the hydrocarbon patterns of coastal surface waters south of Martha's Vineyard reported by Boehm (1977). Therefore, it is unlikely the traces of hydrocarbons found in the sediments at the bow section in July of 1977 came from the *Argo Merchant* cargo. These hydrocarbons could have been from non-cargo sources of oil from the *Argo Merchant* (i.e. hydraulic fluids, etc.) or from non-*Argo Merchant* sources altogether. With the exception of Station 70, the rest of the samples gave no evidence of anthropogenic hydrocarbons, weathered or otherwise, above the normal background levels.

Since the fishing vessel had a draft of only 2 m, it was possible to collect sediment samples on top of the shoals, between the bow and stern section locations, a feat not possible with R/V *Endeavor* (draft of over 5 m). Stations 76(1), 76(2), and 76(3) represent a transect across the shoals from the bow site to the stern site. It was felt that sediments at these stations might have been heavily contaminated with oil as the bow section drifted over them before it was sunk (Grose & Mattson, 1977). In July, no evidence of such contamination was found. Again because of the turbulence of the area, the oil could have been degraded, buried, transported away from the shoals, and/or dislodged into the water column especially since seven months had passed from the time of the original spill.

No evidence of either transportation to other adjacent stations (50, 61, 72) or burial at the wreck site was found. Again, since we obtained sediments only as deep as 10 cm, it is unwarranted to make conclusions based on such samples when daily fluctuations in sediments of two metres are possible.

In summary, the hydrocarbon concentrations in the surface sediments in the vicinity of the *Argo Merchant* wreck site in July 1977, were significantly lower than found on the earlier cruise in February. At only one station was evidence of anthropogenic hydrocarbon contamination found, and these hydrocarbons did not match the *Argo Merchant* cargo oil.

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